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13. ABSTRACT *(Maximum 200 words)*

Collectively the three projects continue to aim at enhancing the science base to make food processing and food quality more predictable. The research effort clearly while dynamically changing to meet the new requirements of food processing aims at developing increased control over product manufacturing issues resulting from variable raw materials. By understanding how variable chemistry affects transport properties and by simulating the effect of changing transport properties on functionality, we are developing the necessary links to predict quality in the presence of variability. The transport models are pointing out to us the key processing and ingredient parameters which need to be adequately controlled to obtain reproducible quality and our in-line sensing research effort is developing the science and the technology to allow rigorous and intelligent control of the food processes. Finally, by understanding how deteriorative reactions in food are a function of the state and composition of foods as well as storage conditions (which in turn affect the equilibrium phase conditions) we are also developing the science base to keep food products stable during storage.

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Food Stability and Shelf-Life

Final Report

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1. Forward

The research effort carried out in this project leveraged ARO funds with funds from a group of industrial firms and from the state of New Jersey to provide a total investment in excess of $1.0 million annually. the ARO funds were key to providing the U.S. Army Natick Research, Development and Engineering Laboratories access to the results of the research. The research approach was to study phenomena in well characterized models, gain understanding of underlying mechanisms rather than macro consequences and translate dthe findings to more complex systems and ultimately to foods.

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4. Body of Report

4.A. Statement of the Problem Studied

The broad goal of the research in the CAFT Basic Research program is to develop molecular understanding of the transformations that food materials undergo during processing and storage so as to generate quantitative design principles to improve current technology. This in turn would enable the manufacturing sector to produce processed foods with optimum quality and storage stability. Such quantitative and thorough knowledge will permit optimization of the design of process equipment, proper selection of operating conditions, and raw materials to produce final products with the desired quality and close tolerances. The research is also aiming at developing new information to optimize storage conditions to maintain food product stability. The inherent variability in the raw materials which are of biological origin and are subjected to variable storage regimes before being processed are clearly a key component of the problem. Primary opportunities for reduction of variability effects are in raw material blending and in processing parameter adjustment. It is, therefore, necessary to understand the chemical changes of constituent species and properties of the materials and products so that appropriate adjustments can be made to fine-tune the process and thus gain close control over end product functionality and performance. This understanding is also needed if stability of the finished products is to be maintained during storage and distribution when texture, flavor, color, hydration potential are subject to change.

Control is achieved by developing quantitative design principles which emerge from simulation of transport phenomena. The transport properties needed are moisture and thermal diffusivity, rheological properties, heat capacities, heat of hydration, gelatinization, etc. The goal of the program is to build on the knowledge base developed in the last three years which allowed us to develop state diagrams for key raw materials, corn and wheat flours and their starch, protein and lipid components. We now want to clearly link transport and physical properties which are dependent upon the state or phase of the material to the various states identified in our state diagrams. The phase of the material is influenced by reactions and order-disorder transitions which occur during processing. It is, therefore, necessary to develop a predictive base for these phenomena during process and after manufacture. These phenomena will then affect texture, color, flavor, safety and nutritional quality of the products. This integrated vision is shown in Figure A.

The program is organized in three key projects. These are the Processed Food Stabilization Project, the Physical Forces Project and the In-Line Sensors Project. The goals of these projects can be summarized was follows:

4.B. Summary of the Most Important Results

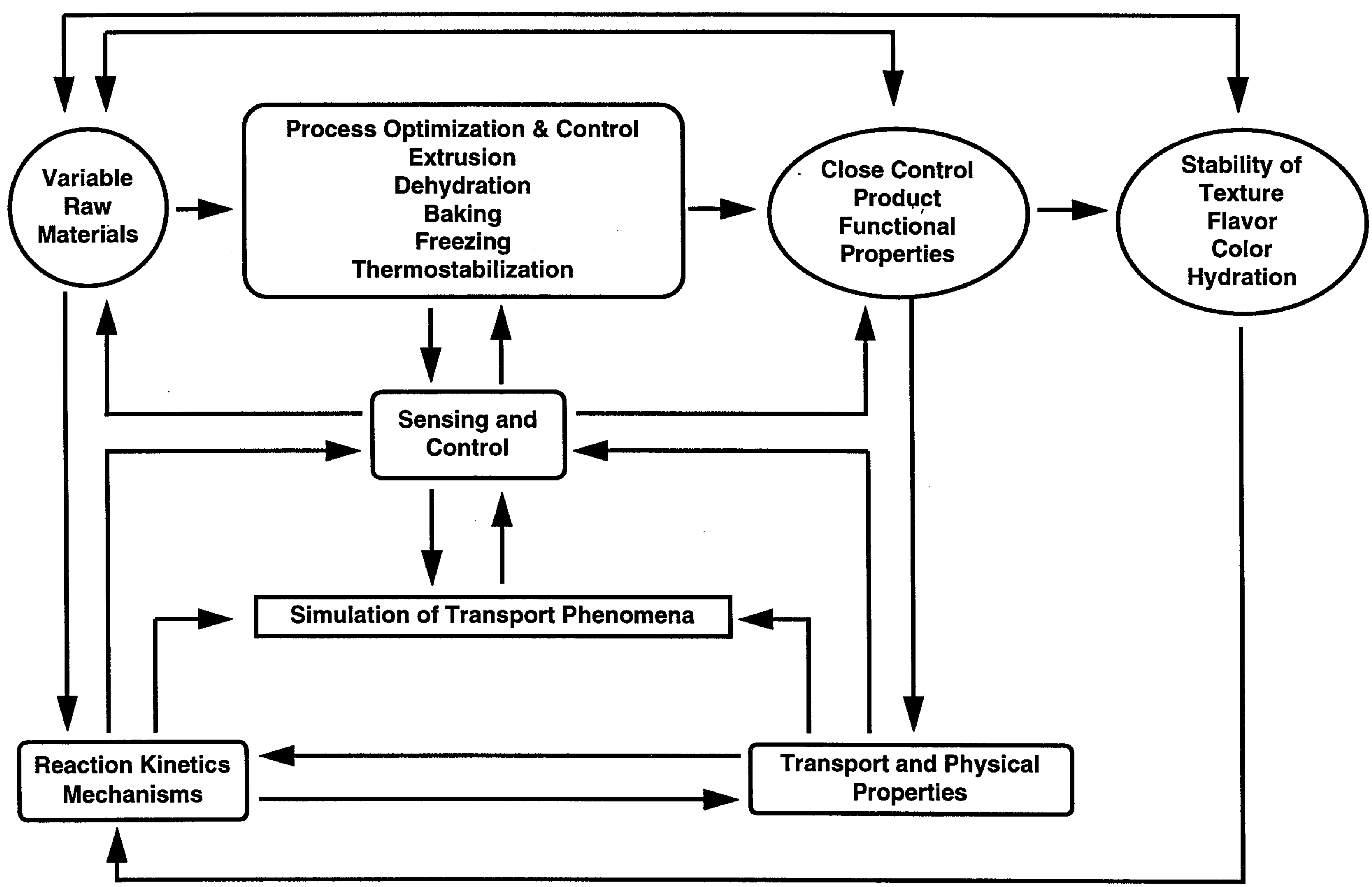
Processed Food Stabilization

The Process Food Stabilization Project is aimed at understanding the phenomena involved in keeping processed foods fresh-like during storage and distribution. The goal will be achieved by gaining physical and chemical understanding of the molecular level phenomena which underlie the fundamental food quality attributes of flavor, color and texture.

Research is focused in the areas of stabilization of flavor, stabilization of texture, stabilization of color, heat/moisture transfer model development including food deterioration kinetics during shelf life to predict changes in stability of food attributes and explorations of high pressure and smart packaging technologies to keep processed foods fresh-like.

Figure A

CAFT Research Program



Physical Forces in Food Systems

Physical Forces in Food Systems project focuses on optimization of flavor and texture of foods during extrusion, microwave, and baking processes. The research is producing strategies, based upon physico-chemical understanding and quantitative predictive model development, which offer product improvements and new product opportunities. Extrusion experiments are carried out using wheat, rice, and potato flours. Mathematical models are being enhance by incorporating kinetics of important chemical reactions leading to better prediction of extrudate quality. The microwave processing and novel baking research is directed toward evaluation of food quality relative to conventional processes.

In-Line Sensors for Food Processing

In-Line Sensors Project aims to identify, develop and demonstrate the feasibility of real time, in-line sensors to address measurement technology needed for the control of food processing systems. The project also aims to identify and implement strategies to enhanced the rapid development, testing and commercialization of new sensors that pass the first test of feasibility.

This document summarizes research findings during September 1992 - September 1995.

4.B.1 Processed Food Stabilization

4.B.1.1 Phase Transitions and their Effect on Chemical Reactivity

Stabilization of processed foods requires understanding of water mediated phase transitions in foods such as the glass to rubber transition which results in enhancement of the mobility of food molecules. Increase in mobility is then coupled with an increase in reactivity since reacting species are now more readily able to react with one another. Many of these reactions affect food quality adversely and therefore control of phase transitions can result in the slowing down of deteriorative reactions thus increasing shelf-life of food products.

In an attempt to understand the effect of the glass transition in browning of foods, the rate of browning in a PVP/xylose/lysine model was studied as a function of T-Tg (where T is the ambient temperature, and Tg the glass transition temperature. It was found that browning rates were affected by temperature, T-Tg and moisture in a complex manner (Karmas and Karel, 1994). Below Tg, the reaction rate was minimal. Above Tg, reaction rate increased with temperature. When crystallization of the matrix (which can occur above Tg) caused an abrupt increase of the browning caused by the reaction between xylose and lysine which were excluded from the crystalline matrix and therefore exhibited an increase in local concentration. When the crystallization was inhibited the increase in browning would also disappear (Buera and Karel, 1994). In further experiments the browning of xylose and lysine in a raffinose matrix was studied. In this system the moisture content had little or no effect on browning. The observed behavior was thought to be due to the partial crystallization of raffinose as its pentahydrate above a critical range of water contents.

We have also used phosphorescence studies to gain further insights about the glass to rubber transitions (Shah and Ludescher, 1993). Phosphorescence studies of surface hydration indicate the protein is in a rigid, perhaps glassy state at low water content but in a flexible perhaps rubbery state at full hydration. Recent DSC studies indicate a well-defined glass transition for lysozyme; the transition temperature is highly sensitive to hydration levels. This is a novel finding for folded, globular proteins. Several phosphorescent probes have been used which show the glass transition in carbohydrate mixtures.

Further, phosphorescence studies of the glassy state of sugar and proteins suggest that, at the molecular level, not all glasses are equal (Shah and Ludescher, 1995). At room temperature sugar glasses appear to be more rigid than those formed in proteins while the glass state of native globular proteins may be more rigid than those of amorphous polypeptides. The studies also suggest that the

glassy state is actually a complex state of affairs. The glassy state is not the same for proteins and sugars and may even differ within each class of food components. Such information may provide insight into the influence of temperature and hydration on the molecular changes that foods undergo during processing and storage.

Clearly differences in the mobility of molecules would result in differences in collision frequency and therefore initiation of deteriorative processes. Detailed probing with phosphorescence studies allowed us to gain a better insight into variations in mobility in the glassy state. We have then used luminescence spectroscopy to monitor the hydration of proteins and sugars and the influence of this hydration on structure surface properties and dynamics at the molecular level. The red edge techniques were used to monitor the relaxation of the polar groups of the matrix around the excited molecule. This newly developed technique may be used to directly measure the extent of matrix relaxation versus time and provide insight into the mechanisms of molecular motion that provide the basis for chemical reactivity in food systems.

4.B.1.2 Effect of Phase Transitions on Collapse Phenomena

Collapse phenomena are associated with changes in texture during freeze dehydration and with release of encapsulated volatiles in solid matrices. We have shown collapse phenomena to also be associated with phase transitions in foods (Karel et al., 1994 and Karel et al., 1994).

In carbohydrates studies, collapse of amorphous carbohydrate systems (sucrose, raffinose) shows an initial fast decrease in specific volume followed by a later slow decrease. Previous CAFT studies of the release of propanol, an organic marker for flavors and other reactants, showed proportional volume changes (Levi and Karel, 1994). Further studies with maltodextrin showed a fast release of volatile organic compounds associated with collapse of glassy matrix with increasing temperature followed by a slow diffusional release. A predictive model of flavor release linking flavor release to temperature, glass transition temperature, moisture has been developed. Such predictive design principles should aid in the design of drying processes to minimize flavor lost and maintain textural quality of dehydrated products.

4.B.1.3 Molecular Dynamics of the Hydration of Proteins

Conformational transitions which proteins undergo as a result of hydration or water loss have a profound impact on availability of moisture in a food matrix which in turn affects the mobility and reactivity of the food matrix. Computational techniques offer tremendous opportunities to examine conformational transitions in proteins.

We have developed techniques to utilize graphic tools to view the shape, spatial extent and properties of the hydration layer of a protein (Shah et al., 1993). We have developed methodology to generate pictures from of results molecular dynamics calculations. While the ultimate goal is to accomplish this for simulations of solvated proteins, the techniques have initially been applied to alanine dipeptide in water. The simulation box is partitioned into an array of cells and properties are calculated for water in each cell. The system can then be visualized by taking "slices" through the box and color coding the water molecules according to the variation in the property of interest (cutting plane), or by looking at a specific value of the property around the entire solute (isosurface). The quantities which have been examined are number density, dipole magnitude and dipole direction. It was shown that the release of water of hydration contributes a term to the entropy which favors folding owing to their greater freedom of movement upon expulsion into the bulk solution (Mutabayashi et al., 1994).

To generate experimental evidence to supplement computational studies, the release of water in protein folding was studied. In the fast step a molecule of lysozyme releases 1265 water molecules. The weight of water released exceeds that of the protein which refolds. Clearly in protein containing foods, the amount of water released when protein refolds will be substantial and would have a marked

effect on texture, flavor retention and the initiation and propagation of deteriorative reactions affecting quality.

As a model of nonpolar interactions in a protein in solution, the methane in water system has been utilized in the development of molecular simulation methodology. Since the structure of proteins and the interactions between proteins and water are instrumental in determining their functional performance in food systems, these calculations allow us to obtain information about protein hydration to aid in the design of proteins with specific functional attributes.

We are currently continuing the computational molecular dynamics studies to understand the effect of high pressure and solvent environment on the properties of proteins. We have also started examining the effect of pressure on chemical properties of various food compounds. In particular, a significant enhancement on the tetramethylpyrazine (TMP) formation at high pressure was observed in the 3-hydroxy-2-butanone/ammonium acetate model system. In a water system, an activation volume of TMP formation under high pressure was found to be -6.82 ml/mole. A mechanism was proposed to elucidate the formation of TMP in a weak acidic condition and high hydrostatic pressure. Solvents such as PG, glycerol, methanol, ethanol, propanol and butanol were found to enhance the TMP formation. Kinetic analyses indicated that TMP formation in aqueous 80% PG and ethanol systems followed pseudo-zero-order reaction kinetics. Since high pressure preservation is of particular interest to U.S. Army Natick RD&E Center as a means to preserve foods without the use of thermal processing which often results in severe treatment of the food these studies will provide useful guidelines to predict, in particular, the reactivity of enzymes which affect food quality so dramatically.

4.B.1.4 Mechanistic Understanding of Flavor Development Through Maillard Reactions

Deamidation of various food proteins was previously shown to be associated with pyrazine formation. Pyrazines are responsible for toasted flavors in food processing operations such as baking and extrusion.

Protein deamidation studies for lysozyme and soy protein have shown that their deamidation is more structurally sensitive at neutral to alkaline pH's (Ho et al., 1994). The extent of deamidation of casein, soy, lysozyme and gliadin over a wide range of moisture contents was also determined. Maximum deamidation for all was at a limited and not at the highest moisture content.

Deamidation studies were followed with systematic studies of pyrazine formation (Hwang et al., 1995). The contribution of ε-amino group of lysine to the pyrazine formation in Maillard reaction was studied by using lysine labeled with 15N at the ε-amino group. The results showed that ε-amino group of lysine contributed significantly to the formation of pyrazine. This suggests that protein-bound lysine residues will still be able to undergo Maillard reaction and lead to the formation of organoleptically important pyrazine compounds.

In the presence of glycine, glutamine and glutamic acid showed the least contributions to formation of pyrazines whereas asparagine had the highest contribution. It was also found that lysine was able to increase the reactivity of glycine, however, arginine could decrease the capability of glycine to produce pyrazines. The variety and quantity of the pyrazine formation depended on the reactivity and type of amino acid used. Since pyrazines are important flavor contributors in baked and toasted foods, suitable amino acids can be used as precursors to enhance the desirable flavor of processed foods.

4.B.1.5 Emulsion Studies

The research discussed so far aimed at developing design principles for solid foods. In this project model emulsion systems aim at developing design guidelines for the stability of several liquid and semi-solid food systems such as mayonnaise, salad dressings, microemulsion type soft drinks, etc.

Initially these studies focused on model four component microemulsion series (cetylthimetrylammonium bromide/hexadecane/butyl alcohol/water) (Yao and Romsted, 1995). The research focused on monitoring interfacial composition and binding constants over a wide range of water/hexadecane ratios. It was established that the hydrophobic arenediazonium salt reacts with the -OH head groups of nonionic surfactants in competition with water. Results with BuOH in four component microemulsions show that it is possible to determine the location of any molecule, e.g. a flavor component, in a microemulsion and possibly emulsion system that has a weakly basic functional group which reacts with the arenediazonium salt probe. Results with nonionic surfactants suggest that it should be possible to correlate interfacial water concentration with the stabilities of nonionic micelles and microemulsions. Furthermore, estimates of interfacial water concentrations from dediazoniation reactions indicate that characteristic interfacial water concentrations correlate with structural transitions in cationic microemulsions and nonionic micelles and therefore aggregate stability. Peptide interactions with surfactant aggregates in bulk solution and moonlighters should provide new information on the mode of binding of proteins to aggregate interfaces and how proteins act as emulsifiers. In principle, the dediazoniation probe technique can be used with more complex systems containing mixtures of emulsifiers such as mono- and diglycerides, alkylcarboxylates, alkylphosphates and even phospholipids. To complement the surface composition studies, new studies on probing the relationship between emulsion stability, interfacial composition and properties and protein adsorption were initiated. Accomplishments include the following: (i) the characterization of the surface dilational elasticity and surface adsorption of a model protein layer at the air/water interface; (ii) the design of a fluid droplet coalescence life-time experiments; (iii) the successful illustration of the correlation between coalescence half-time and interfacial dilational elasticity; and (iv) the design and construction of a modified Langmuir through to measure the dilational elasticity of protein covered oil/water interfaces.

The results obtained from surface dilational elasticity measurements and the success obtained in correlating them with droplet half coalescence life times when combined with surface compositional measurement studies are leading to rules of selection of surface active materials for optimal stability in emulsion products.

4.B.1.6 Mathematical Simulation of Hygrostress Formation During Dehydration and its Effect on Quality

Parametric analyses were performed for thermo- and hygrophysical changes in viscoelastic food of solid or hollow cylinder shape undergoing drying (Itaya et al., 1994). According to the analyses, stress crack formation and propagation may be controlled by lowering initial food moisture and/or by increasing air relative humidity. The influence of operational and physical parameters on hygrostress formation in plate-shaped or spherical elastic food was examined qualitatively applying the simplified method developed. The globally maximum tensile stress occurs always on the exposed surface during a drying process and at the center during a hydrating process.

In the drying of an infinite cylinder, a semolina pasta model, no cracks were formed when the initial system moisture was below a critical value. As the cylinder wall thickness increased, crack density decreased but crack size increased. These findings provide insight into the control of parameters to avoid crack formation.

The nonisothermal hygrostress analyses show approaches to minimize stress crack formation and propagation in viscoelastic food of a solid or hollow cylinder shape.

We have also developed simplified easy-to-use method for maximum hygrostress formation in elastic food of a solid cylinder and obtained sample application results. This simplified method is easy and simple to use for estimating maximum hygrostress formation in cylindrical elastic food.

4.B.1.7 Inhibition of Enzymatic Browning in Potato

Browning of raw fruits, vegetables and beverages is a major problem in the food industry and is believed to be one of the main causes of quality loss during postharvest handling and processing. The cutting of certain plant tissues such as banana, apple and potatoes results in rapid color development which renders the product unappetizing in appearance. The storage life of peeled or cut raw potatoes is limited by the onset of enzymatic browning. Browning is more severe when the food has been subjected to processing. It can result from cutting, peeling, pureeing, pitting, pulping, freezing or dehydration. In potato tubers, injury during mechanical harvesting and subsequent handling causes areas of the tuber to develop a discoloration called blackspot. The biochemical pathway leading to browning in potato is not well understood. Polyphenol oxidase (PPO) catalyzes the oxidation of mono- and diphenols to o-quinones, which can polymerize spontaneously by further nonenzymatic reactions to form the brown pigment, melanin, which is produced during browning. Potato cultivars differ in their susceptibility to enzymatic browning. Russet burbank, which is the major commercial potato cultivar in the U.S. is very susceptible to browning. In addition to PPO levels and activity, other factors, such as the availability of the substrates and the dry matter content of the tuber have been proposed to play a role in enzymatic browning in potato. We hypothesize that PPO is the key enzyme regulating enzymatic browning in Russet Burbank. We will directly test this hypothesis by inhibiting PPO activity in Russet Burbank tubers by expressing antisense RNA. We have isolated cDNA clones encoding PPO from potato and tomato and constructed plant expression vectors containing these cDNAs in sense and antisense orientation. These constructs are currently being transformed into Russet Burbank potato. We Will analyze PPO levels and activity in transgenic tubers and evaluate the susceptibility of these tubers to enzymatic browning. If browning can be inhibited in Russet Burbank tubers without any detrimental side effects, it will be possible to utilize similar approaches to prevent enzymatic browning in a wide variety of food crops without applying inhibitors, such as sulfating agents or heating.

4.B.2 Physical Forces in Food Systems

The Physical Forces Project made substantial progress in developing quantitative understanding of the physico-chemical changes occurring during extrusion of starch based food materials.

4.B.2.1 Numerical Simulation of Extrusion

One of the key goals of the project is to develop a prediction of the transport phenomena using numerical simulation. A numerical model for a single-screw extruder that takes axial diffusion into account was developed and validated by comparison with the experimental results (Chiruvella et al, 1993). A simplified model for the solid conveying zone was developed and incorporated into the numerical simulation. A finite volume scheme was developed to model the intermeshing zone of a twin-screw extruder. This was linked with the translation zone to yield the simulation of transport phenomena in an entire twin-screw extruder (Sastrohartono et al., 1994).

4.B.2.2 Experimental Studies

To validate the numerical simulation and to provide data to support the numerical calculations, several experiments have been conducted. First, the single-screw extruder was modified to permit the barrel of the extruder to be removed from the screw within 7 seconds after the extruder was suddenly stopped. It was found that for corn meal full conversion was reached in less than 10 cm of the barrel length for most cases. The conversion reaction rate was zeroth order (Wang, 1993).

To understand transport in the powdery zone, starch powder was extruded using a single screw extruder. It was found that the powder initially compacts to a raw mass and then converts near the end of the extruder. Heating plays a very significant role in compaction and in start of conversion, making the rheological region essentially equal to the region over which the fluid exceeds the gelatinization,

or conversion, temperature (Zheng and Wang, 1994). This information must be taken into account in the overall transport phenomena, in order to design and optimize the overall extrusion process.

To begin to understand the role of reverse screws on the operation of twin screw extruders, the effect of reverse screws on the conversion of starch and the residence time distribution was investigated at low (<100°C) temperatures (Gogoi and Yam, 1994). Quick stop runs were used to examine the state of starch before and after the reverse screw element. Reverse screws had a profound impact on the residence time distribution.

To generate data for validation of models and scale-up strategies, extrusion experiments were carried out on the ZSK-30 twin screw extruder to determine overall energy balance and the convective heat transfer coefficient between the barrel surface and the extrudate. The measured heat transfer coefficients were found to be strongly related to process moisture and barrel temperature. Velocity distributions in the screw channels of ZSK-30 were measured using laser Doppler anemometry. Comparisons with predicted velocity distribution showed good agreement in the translation region (Karwe and Sernas; 1995).

4.B.2.3 Post Extrusion Transport Phenomena

A key component of the operation of extruders is the phenomena involved in generating quality of extrudates once the extrudate exits the extruder. In post-extrusion experiments, it was found that the temperature of the extrudate dropped more rapidly as the process moisture content increased. The rate of cooling was not significantly affected by the throughput. The rate of moisture loss from the extrudate increased when the die temperature increased. These findings are enabling us to develop quantitative calculations of the properties of extrudates involved in expansion, such as viscosity.

4.B.2.4 Kinetic Studies

Numerical calculations need kinetic equations which are relevant to extrusion. The applicability of a shear induced starch conversion in a twin-screw extruder was studied using corn meal. It was found to follow a zero order reaction (Yam et al., 1994)

The kinetic parameters of the tribological conversions for amioca starch were obtained. The tribological conversions were found to be more sensitive to shear forces than that of rheological conversion. The transition from tribological zone to rheological zone was material dependent, in addition to water content, percent conversion, and pressure.

4.B.2.5 Measurement and Simulation of Rheological Properties

Extruded food materials undergo several order-disorder transitions and chemical reactions which influence physical properties such as rheological properties and thermal as well as mass diffusivities. State diagrams for zein and gliadin were developed showing various phase boundaries as a function of temperature and moisture content (Madeka and Kokini, 1994). The glass transition could be predicted by Gordon-Taylor equation. Domains of entangled polymer flow, aggregation, softening and reactive phases were identified (Kokini et al., 1994).

To provide rheological data at room temperature, we studied the viscoelastic properties of wheat flour doughs. In the simulation of rheological properties of wheat flour doughs and their components, a linear memory function was used for wheat flour doughs which can be expressed by a Maxwell model (Huang and Kokini, 1994) The non-linear rheological properties could be accurately predicted by the Wagner model.

In the free flowing region during extrusion, we measured viscosity of wheat flour extrudates. The viscosity of wheat flour extrudate (40% moisture, 98°C) approximately followed a power function relationship with specific mechanical energy (SME) and shear rate. Viscosity decreased with an increase in SME.

To predict loaf volume during baking or extrudate expansion, biaxial extensional viscosities obtained from RSA-II were superimposed on those from squeezing flow method. Biaxial extensional viscosities obtained using the bubble inflation method were much larger than those obtained using squeezing flow (Huang and Kokini, 1993) Biaxial extensional viscosities showed a power-law relationship with extensional rate for both lubricated squeeze film method and bubble inflation method. An equation to predict gas cell expansion during isothermal fermentation was developed. The predicted values of dough volume expansion agreed with data in literature.

4.B.2.6 Mixing Studies

Mixing is a key component of extrusion,; it is also, independently of extrusion, a key unit operation of the food industry. Our mixing studies aim at making selection of mixers as well as their scale-up easier. To develop quantitative understanding of the mixing phenomena in the Farinograph, six planes were measured for velocity data (using Laser-Doppler Anemometry) at equally spaced grid points for three model working fluids. This information leads to an understanding of the effectiveness of mixing in the Farinograph.

4.B.2.7 Thermal Properties Measurement

Correlation equations for specific heat of wheat flour and thermal conductivity of amioca starch as a function of temperature and moisture content were developed. This information was incorporated into the mathematical models simulating extrusion transport phenomena.

Pressure variable differential scanning calorimetry revealed the temperature of the thermally-induced transitions (melting, glass transition) to be highly dependent on moisture content, while remaining relatively independent of pressure up to 60 atm.

4.B.2.8 Determination of Fragmentation in Cereal Materials

It was found that the extent of fragmentation, as monitored by reduction in the Tg of the extrudates, correlated with the specific mechanical energy (SME) input (Kaletunc and Breslauer, 1993). The Tg of extrudates correlated with two important sensory textural properties namely, crispness and denseness.

We also monitored fragmentation using gel permeation chromatography and methylation analysis. The interaction of low moisture (16%) and low temperature (160°C) had a significant effect on the reduction in weight average molecular weight (MW) values of wheat flour following extrusion. Qualitative results showed that there was a reduction in the number of branches averaged over the molecular weight distribution of the polymers (Politz et al., 1994).

In extrusion-induced starch fragmentation in wheat, fragmentation was most pronounced in amylopectins. Methylation analysis showed only modest changes in linkage distributions. The lack of dextrins of oligosaccharides suggests that fragmentation occurs primarily in the B chains of amylopectin.

4.B.2.9 Determination of the Origin of Cross-Link Formation During Extrusion

In wheat flour extrudates, when disulfide contents were high, distinctive individual gliadin components were lost, but the gliadin fractional absorbance and weight distribution were largely retained. Hardness and gumminess scores were high in these samples, and free radical signals were small. Hardness scores were lower than with high S-S samples, but no other textural characteristics appeared to be related to high -SH. The high -SH samples did show the greatest starch fragmentation, however, and EPR signals of free radicals were strong, with substantial contributions from various sulfur-centered radical species.

EPR signals from wheat flour extrudates varied both quantitatively and qualitatively with extrusion conditions and protein content of the flour. Under the conditions of extrusion selected, four types of

radicals were formed: nitrogen-centered radicals on the protein backbone and side chains, thiol (-S•) radicals, disulfide radicals (-S • S-), and sulfoxyl (-SO•) or protein peroxyl (-ROO•) radicals. High stable free radical populations correlated with increased sulfhydryl contents, increased evidence of protein scission and crosslinking by HPLC and PAGE, high puff ratios and low volatile retention. In contrast, extrudates with increased disulfide contents had low intensity EPR signals with little or no evidence of sulfur radicals, little evidence of macromolecular alteration by HPLC, tight gummy texture with little puff, and high volatile retention.

The effect of extrusion temperature on changes of extractable proteins in wheat flour (W) and the mixture of wheat flour and egg white (WE) were studied to elucidate the mechanisms concerning protein solubility and disulfide bond breaking and/or forming. The egg white was easier to denature and/or aggregate than wheat flour protein during extrusion. It was found that the total content of the disulfide bond in the extracts of extrudates decreased as the extrusion temperature increased. It was also found that the protein molecules containing higher levels of cysteine/cystine in wheat flour and egg white denatured easily by extrusion. The water-holding capacity of all extrudates was more than twice that of nonextruded samples. The expansion ratio of the WE-extrudate was significantly lower than that of the W-extrudates. The foam stability of WE-extrudate increased with increase in extrusion temperature.

4.B.2.10 Effect of Free Amino Acid on Texture and Flavor Control

To understand the effect of free amino acid on the texture and flavor of extrudates, extrusion experiments were carried out on the ZSK-30 using wheat flour with added cysteine. Since cysteine acts as free radical scavenger, it reacts with the nitrogen and sulfur based free radicals formed during extrusion, which results in disulfide bonds breaking and forming resulting in textural changes of the extrudate. With addition of cysteine during extrusion, the expansion ratio of extrudate decreased, the bulk density decreased and the water-holding capacity decreased systematically. Higher water holding capacity indicates puffier and lighter structure of extrudate which is a desirable characteristic. Addition of cysteine to wheat flour during extrusion reduced bulk density of extrudates, increased gumminess and cohesiveness, decreased fracturability and made the product whiter. The cut extrudates puffed through the cut surface making them popcorn-like. Microscopic examination showed that added cysteine decreased the cell size, made the cell size distribution more even and made the cell walls thinner by about 90%. EPR studies indicated that addition of cysteine reduced free radical content of extrudates preferentially affecting the nitrogen-centered radicals produced during scission of peptide chains.

Cysteine also affected the flavor profile of extrudates considerably. Addition of cysteine during extrusion generated thiophenes and thiazoles which have meaty flavor notes and also pyrazines and pyrroles which have toasted flavor. Cysteine also reduced the undesirable lipid degradation products.

4.B.2.11 Volatile Flavor Development During Extrusion

The volatile compounds of wheat flakes prepared by extrusion cooking were compared to those of conventional pressure cooking. The volatiles generated from the browning reaction in the pressure cooked product were about four times higher than those in the extruded product (Huang et al., 1994).

It was found that important flavor compounds, such as pyrazines, could be generated by the reaction of glucose with hydrolyzed wheat gluten (HWG) and deamidated hydrolyzed wheat gluten (DHWG). The aroma of both reaction mixtures could be described as nutty and toasted. The aroma of the reaction derived from the DHWG was much stronger than that of HWG. Quantitative analysis also indicated that DHWG produced 19 times more pyrazines than HWG.

During extrusion, higher amylopectin levels in starch resulted in increased amount of condensate collected at the die. High amylose content resulted in increased moisture retention in extrudates. At

least 7 substituted pyrazines were present in the high amylopectin containing samples. The contribution of amino and amide nitrogen atoms to pyrazine formation in both dry and liquid systems was investigated. The results indicated that deamidation did participate in pyrazine production and more than half of the nitrogen sources of pyrazines came from the amide side chains of glutamine. The yields of pyrazines from the dry system were higher than that from the liquid system.

In order to improve the flavor of extruded products, a precursor (1% of hydrolyzed wheat gluten) was added during extrusion of wheat flour. It was found that the precursor reacted with the reducing sugars to produce desirable flavor compounds such as pyrazines which give toasted aroma. The addition of the precursor also suppressed the formation of undesirable lipid oxidation products.

A model system consisting of 89% Amioca, 5% lysine and 6% glucose was used to measure reaction kinetics parameters of flavor formation. Pyrazines were selected as key flavor compounds (Huang et al., 1994) An internal standard (pyramidene) was used to verify the recovery of the volatiles, which was close to 100%.

4.B.3 In-Line Sensors

Progress in In-Line Sensors research focused on two key areas: 1) development of new sensors in areas identified as being important to CAFT's industrial members and demonstration of feasibility of real-time in process measurement with prototype system designs and 2) trials toward validation of commercial potential, identification of able instrument manufacturers, and facilitation of implementation strategies with CAFT's industrial member companies.

Cooperative projects with several instrument manufacturers have continued toward preparation of prototype moisture sensor systems for field trials with member companies. They include: the porous fiber optic sensor, thermomoisture sensor, and fluorescent film sensor. New development was initiated for prototype piezoelectric moisture sensors. These activities are described below. In addition, a prototype screw pump rheometer has been demonstrated on CAFT's Werner and Pfleiderer pilot plant extruder and is available for member trials.

Sensor research initiatives in on-line pH sensing and flavor/aroma sensing are progressing toward system feasibility demonstration. New research studies are underway for sol-gel type fiber optic chemical sensing and in biosensing for triglyceride determination. These accomplishments will be described in detail below.

4.B.3.1 Moisture Sensors

4.B.3.1.1 Thermomoisture Sensor

Studies in relation to CAFT's thermomoisture sensor have been focused on the next generation of sensors to simplify fabrication and detection techniques. Based on mathematical models that have been developed (Voudouris and Hayakawa, 1994), a miniaturized probe has been developed. Development of a prototype the CAFT patented miniaturized thermomoisture sensor is being done with Schutz Engineering Corp., assisted by funding through the Technology Transfer Merit Program (TTMP) of the New Jersey Commission on Science and Technology. The system incorporates a miniaturized electronic power pack and data acquisition coupled to a notebook computer. From tests of high-amylopectin corn starch gels with dissolved sucrose, performance equations have been developed that will allow rapid moisture determination by computer program. Correlations for a number of other different food systems are currently being developed so that field trials of the sensor in member company pilot plants can be undertaken. Several CAFT member companies have expressed interest in such sensor trials with prototype systems.

4.B.3.1.2 Porous Fiber Optic Sensors

Research in this area focused on continuing to improve the performance of the sensor in both low and high temperature prototype sensors. Characterization of the low temperature system has established the calibration curve and operating range for the porous Fiber optic system. Additionally, a new data acquisition system has been constructed in conjunction with Pro Optical Technologies. The new, simplified, field-ready systems uses a single wavelength for both measurement and as a reference wavelength. Initial trials have shown good performance at temperatures up to 80°C and moisture levels between 45-75%. A high temperature material (up to 300°C) has been prepared and showed good response and moisture correlation in laboratory apparatus.

Schutz Engineering was commissioned for this sensor as well, to build a new field usable prototype measurement systems. Subsequently RoMarck Co., Maplewood, NJ, was brought in to cooperate with Schutz Engineering and a new probe configuration of the sensor has also been fabricated. A field prototype system has been constructed to be tested at a member company facility. The system's components were evaluated in CAFT's sensor test chamber which will be described below. The sensor has also been successfully tested in the CAFT's jet-sweep oven.

4.B.3.1.3 Ceramic Sensors

The laboratory-scale ceramic moisture sensing prototype previously developed has been redesigned using newly available highly stable MACOR™ ceramic as a probe material for the sensor housing design for the sensor pellet material. The new sensor is 12 inches long and 6 inches in diameter. The prototype eliminates the bulky auxiliary nitrogen cooling system. Also a new low frequency (10Hz) impedance measurement circuit was designed and built and optimization studies were conducted. Successful testing has occurred at gas temperatures up to 300°C (450°C pellet sensor temperature). For long term high temperature measurement, new electrodes were designed, and fabricated. Experiments with this design were more stable and reproducible than the previous approach. However, to make the sensor even more user friendly, a new and smaller prototype sensor which incorporates a heater circuit etched or printed on one side of an inert alumina wafer with the active ceramic sensor film on the other side has been designed and fabricated.

4.B.3.1.4 Inorganic Film Based Moisture Sensors

A new membrane electrode of LiSO3 PEEK (polyphenylether ether ketone) has been fabricated utilizing a commercial interdigitated electrode base. Deposition of the inorganic film, sulfonated polyphenylether ether ketone (PEEK), on gold interdigitated electrodes was optimized to establish reproducible thin films. This sensor has been linked to a newly developed highly sensitive low frequency complex impedance measurement system. The sensing system is expected to be a miniaturized, stand-along system which can be directly interfaced with computerized process control systems. Successful laboratory testing is complete. Furthermore, a sensor probe has been developed with the inorganic film (PEEK) for trials in the CAFT sensor chamber. Where performance tests showed sensitivity and response comparable to commercial low temperature sensors. High temperature tests are underway. In addition, a new polymer material analogue of the PEEK poly(phenyl ether-ether-ketone), with improved high temperature performance properties was identified. Evaluation is in progress. An instrument company has been identified that could commercialize this sensor when proven.

4.B.3.1.5 Piezoelectric Nylon 7 and 11 Sensors

Characterization and preparation of the piezoelectric sensing materials has continued (Scheinbeim and Newman, 1993 and 1994; Scheinbeim et al., 1994). A new polarization method has been uncovered for the odd nylons that significantly simplifies material processing by eliminating any necessity for cold drawing of the nylon film. This discovery should markedly reduce the cost of commercialized sensors.

The performance data have been collected for piezoelectric nylon 7 moisture sensor at temperatures up to 175°C. Characterization tests of piezoelectric nylon 7 moisture sensitive films revealed that molecular orientation during film preparation is critical; films must be drawn (stretched) less than previously believed in order to optimize piezoelectric response. Bilaminate films were also prepared and characterized (Mei et al., 1995) Samples of nylon 5 have been prepared and are currently being tested at temperatures up to 250°C. A prototype Nylon 7 sensor has been prepared for trials in the sensor test chamber.

CAFT has also requested Schutz Engineering Corp. to develop the nylon 7 moisture sensor. Tests which identified surface moisture effects at high humidity resulted in design modifications using guard rings to separate active sensor elements to achieve successful measurement. Currently two sensor probe concepts have been prepared and are being evaluated.

4.B.3.1.6 Fluorescent Film Moisture Sensor

Preliminary field tests of a first generation prototype sensor were carried out identifying areas requiring improvement. New sensor mounting techniques were developed and proven in laboratory trials (Pedersen et al., 1992). These trials showed sensitivity similar to commercial low temperature instruments but with slightly faster response time. A spectral data acquisition system has been developed and fabricated by a local area vendor.

Working with Luz Systems, Inc., to develop a prototype which can be used in plant trials, an electro-optical measurement system was developed. The integrated sensing system has been tested in CAFT's test chamber to about 90°C. These tests have led to characterization data for the test protocol developed by CAFT's industrial monitors.

4.B.3.2 Rheological Sensors

Two stand alone dough rheometers have been designed that offer significant advantage in product development. Single-screw screw pump rheometer performance has been optimized through examining characteristics such as torque, power axial thrust in relation to speed and clearances. Field application of the rheometer has been evaluated by two member companies including a test of production type materials. A strategy for commercial development of the screw pump rheometer for food system application including flow side stream requirement was developed with member companies, CAFT, and the cooperating vendor Rheometrics, Inc.

As a result, a prototype screw pump rheometer has been designed and fabricated. The rheometer has been demonstrated in conjunction with the Werner & Pfleiderer twin screw extruder in CAFT's pilot plant. Modifications were implemented to eliminate seal leakage and further tests were conducted at Stevens Institute of Technology laboratories. The overall system is intended to be used with various highly viscous systems.

Following successful demonstration of the screw pump rheometer (SPR), a corotating twin screw processor/rheometer was designed, built and tested (Gogos et al., 1994). This device can be a stand-alone viscometer which, with interchangeable kneading elements can also act as a process/equipment development tool. Newly available high sensitivity pressure transducers allow broadening the range of viscosity measurement on the SPR.

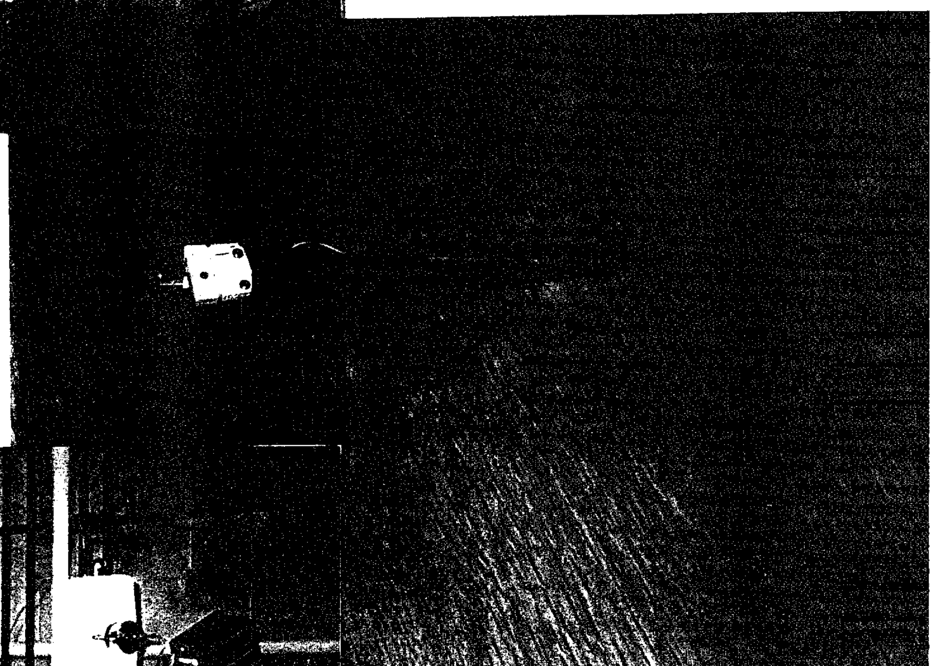
CAFT-developed In-Line Sensors are shown in Figure B.

4.B.3.3 CAFT's Sensor Test Chamber

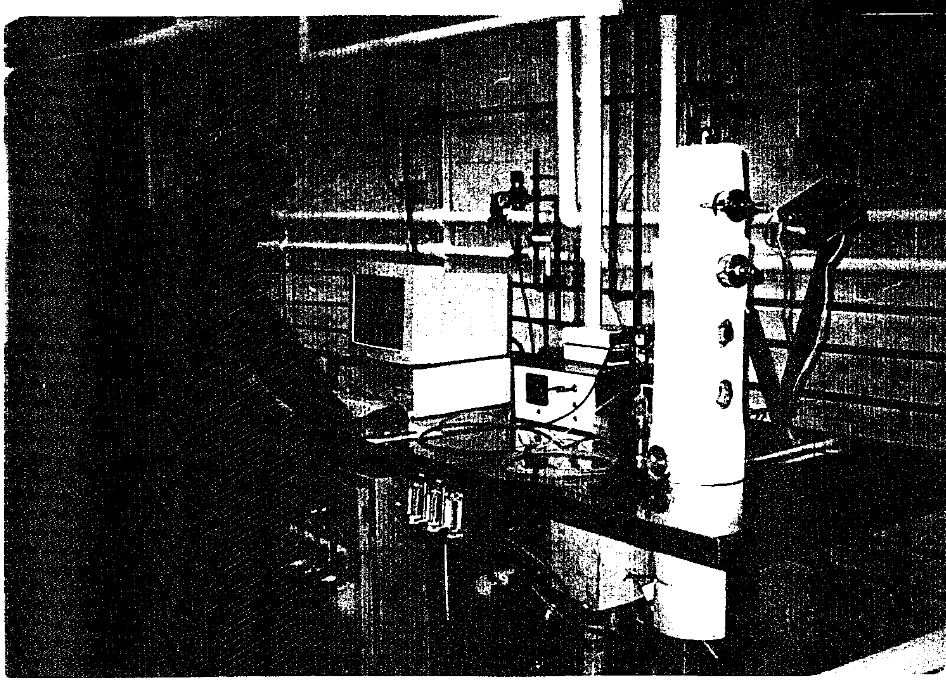
To test the performance of its sensor materials and sensor prototypes, CAFT decided to build a high temperature well controlled test chamber. CAFT's sensor test chamber has been constructed and multistep automation and debugging of the data acquisition and computer control system has been

FIGURE B
CAFT-Developed In-Line Sensors

Porous Fiber Optic Moisture Sensor
Uses: Ovens, dryers at temperatures > 20°C and < 300°C, moisture between 10 and 100%
Faculty Inventor: Dr. Mahmoud Shahriari
Manufacturer: Pro-Optical Technologies, Maplewood, NJ; RoMark, Maplewood, NJ; Schutz Engineering, Summit, NJ

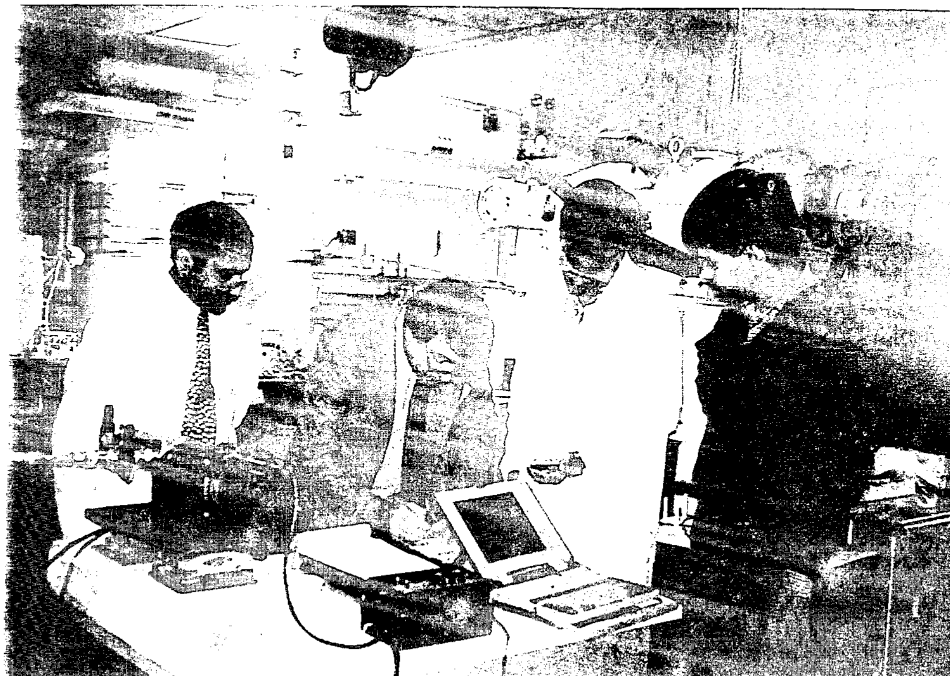


Thermomoisture Sensor
Uses: Viscous, semi-solid, and powder materials at temperatures < 120°C,
Faculty Inventor: Dr. Kan-ichi Hayakawa
Manufacturer: Schutz Engineering, Summit, NJ; Scientific Instrument Services, Ringoes, NJ



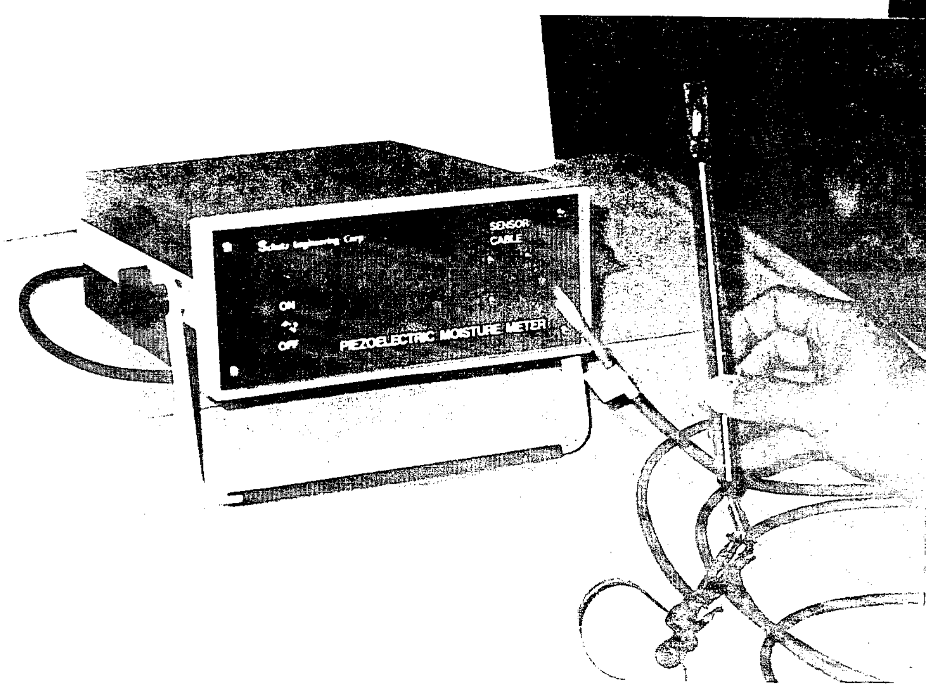
Fluorescent Film Moisture Sensor
Uses: Ovens, dryers at temperatures > 20°C and < 90°C, moisture between 1 and 100%
Faculty Inventor: Dr. Henrik Pedersen
Manufacturer: Luz Systems, Holtsville, NY

Figure B
CAFT-Developed In-Line Sensors



Screw Pump Rheometer
Uses: Viscosity measurements during extrusion, mixing
Faculty Inventor: Dr. Costas Gogos
Manufacturer: Rheometrics, Piscataway, NJ;
Stevens Institute of Technology
Polymer Processing Institute, Hoboken, NJ

Ceramic Moisture Sensor
Uses: Ovens at temperatures > 20°C and < 300°C
moisture between 10 and 100%
Faculty Inventor: Dr. Martha Greenblatt
Manufacturer: not yet selected



Piezoelectric Moisture Sensor
Uses: Ovens, dryers at temperatures > 20°C and < 205°C,
moisture between 2 and 100%
Faculty Inventors: Drs. Jerry Scheinbeim and Brian Newman
Manufacturer: Schutz Engineering, Summit, NJ

completed. In addition to moisture sensing, the chamber can be utilized for other gaseous compounds such as CO, CO_2 and NOx. The chamber allows testing of up to four sensors simultaneously, in environments up to 300°C. At temperatures up to 100°C, the chamber can operate at moisture levels from 0 to 100%. The chamber has been tested over its design range using control algorithms developed in "Workbench" software. Three new sensors are being installed for operating protocol tests in the simulated process food system environment.

4.B.3.4 New Sensors

New sensors for real-time measuring of processing parameters to respond to the changing needs of its industrial member companies, CAFT has introduced several new in-line sensors research areas. These are described below:

4.B.3.4.1 Ceramic pH Sensing Materials

A number of new ceramic pH sensing materials have been examined over a range of pH from 3 to 10 at ambient temperature of 25°C (Wang et al., 1994). Both the molybdenum oxides bronze crystals as well as the samarium-ceria thick membrane responded reproducibly in lab experiments. The oxide bronzes show nearly ideal Nerstian behavior; the ceramic membrane did not show similar linear behavior but is expected to perform better at higher temperatures. Experiments are underway to check cross sensitivity (alcohol, NH_3...) accuracy and stability and to then construct a solid-state pH probe.

4.B.3.4.2 Flavor Sensors

Study of the pyrazine flavor sensor has shown that several ruthenium complexes will bind with pyrazine which can then be detected by fluorescence. Reaction kinetics have been examined to enable selection of a probe material with a fast capture rate to be able to quantify amounts of pyrazine. Experiments with brewed coffee showed considerable enhancement of fluorescent intensity for detection. Discovery of an electro-analytical technique shows promise of discrimination between pyrazines in complex mixture.

4.B.3.4.3 Biosensors for Triglyceride Determination

Preliminary experiments of the biosensor concept for triglyceride determination demonstrated how the triglyceride concentration, extracted from olive oil and mayonnaise samples into cyclohexane, can be coupled to near-infrared spectral analysis for the quantitation of fat content. Also, studies showed that the triglycerides can be removed from the food substance by enzymatic reactions with an immobilized lipase to hydrolyze the fat into organic solvent. This provides a basis for the design of a stand-alone sensing method as the next step in this study.

4.B.3.4.4 Fiber Optic Chemical Sensors

New fiber optic chemical sensors are under development using sol-gel techniques to immobilize suitable indicator compounds which enhance selectivity and sensitivity of the sensor. H_2S is the first important target under study. Durability of the sol-gel films has been studied at room temperatures to 70°C. Initial results indicate leaching does not occur providing one indication measure of safety for use in sensitive food environments.

4.C. Summary

Collectively the three projects continue to aim at enhancing the science base to make food processing and food quality more predictable. The research effort clearly while dynamically changing to meet the new requirements of food processing aims at developing increased control over product manufacturing issues resulting from variable raw materials. By understanding how variable chemistry affects transport

properties and by simulating the effect of changing transport properties on functionality, we are developing the necessary links to predict quality in the presence of variability. The transport models are pointing out to us the key processing and ingredient parameters which need to be adequately controlled to obtain reproducible quality and our in-line sensing research effort is developing the science and the technology to allow rigorous and intelligent control of the food processes. Finally, by understanding how deteriorative reactions in food are a function of the state and composition of foods as well as storage conditions (which in turn affect the equilibrium phase conditions) we are also developing the science base to keep food products stable during storage.

4.D. List of All Participating Scientific Personnel

Name	Degree Earned While on Project
Abib, A.	
Akiyama, T.	
Beaver, M.	M.S.
Breslauer, K.	
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Buera, B.P.	
Chan, E.	
Chan, K.Y.	
Chatterjee, S.	
Chaudhuri, B.	
Chen, C-W.	
Chen, Q.	
Chiew, Y.C.	
Chiruvella, R.V.	Ph.D.
Cho, M.H.	
Cisneros, F.	
Cocero, A.M.	Ph.D.
Cruz, R.	
Cuitino, A.	
Daun, H.	
Davidar, C.	
Detig, C.	
Ding, J.	Ph.D.
Elliott, J.J.	
Esseghir, M.	
Feng, S.	
Fox, S.	
Foygel, K.	
Fu, H-Y.	
Gasparian, G.	
Giannakakos, P.	
Gibson, S.	
Gluck, J.B.	
Godavarti, S.	
Gogoi, B.	Ph.D.
Gogos, C.G.	
Greenblatt, M.	
Hamblin, Jesse	
Hartman, T.G.	
Hayakawa, K.	
Henrikson, F.W.	
Herskowitz, G.J.	
Ho, C-T.	
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Huang, R.M.	
Hwang, H-I.	Ph.D.
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Isied, S.S.	
Izzo, H.	Ph.D.
Jaluria, Y.	
Ji, S.	
Jiang, Q.	
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Kahn, P.	

Name	Degree Earned While on Project	Name	Degree Earned While on Project
Kaletunç, G.		Nakamura, T.	
Karel, M.		Newman, B.A.	
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Karwe, M.V.		Overaker, D.	M.S.
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Kiri, S.		Peterson, M.	
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Kobayashi, T.		Poltiz, M.	Ph.D.
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Lei, M.		Riha, W.	
Lelouche, Isabelle		Robinson, R.	
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Levine, S.		Sable, T.	
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Nair, M.		Sohn, M.	

Name	Degree Earned While on Project
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Tong, C.H.	
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Voudouris, N.	M.S.
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Wang, C.F.	Ph.D.
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Wasserman, B.	
Wen, L.	
Wu, X-F.	Ph.D.
Wur, S-L.	
Yam, K.L.	
Yamshita, K.	
Yao, J.	
Yoo, S.S.	
Yu, T.H.	
Zafar, M.	
Zhang, Y.	
Zhang-Mei, B.	
Zheng, X-G.	Ph.D.
Zheng, X.	
Zhou, L.	

5. Report of Inventions

No patents issued.

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